

WHAT IS CLAIMED IS:

1. A method for producing pulp and lignin from lignocellulosic material, the pulp comprising cellulose, the method comprising:

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(a) contacting the lignocellulosic material with an aqueous acid solution to impregnate the lignocellulosic material, the aqueous acid solution comprising from about 10% to about 40% by weight of the acid;

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(b) heating the lignocellulosic material in two stages, the first heating stage being carried out for a period of time which is sufficient to depolymerize lignin within the lignocellulosic material without substantially degrading the cellulose or lignin in the lignocellulosic material, the second heating stage being carried out at or above the boiling point of the acid to distill off the acid;

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(c) contacting the lignocellulosic material with an aqueous alkaline solution under heat to solubilize lignin in the alkaline solution, leaving a black liquor;

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(d) removing the pulp from the black liquor;

(e) adding sufficient acid to the black liquor to precipitate the lignin; and

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(f) removing the lignin from the liquor.

2. A method according to claim 1 wherein the acid is nitric acid.

3. A method according to claim 1 wherein the lignocellulosic material is

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contacted with the aqueous acid solution in step (a) for at least 30 minutes.

4. A method according to claim 3 wherein the lignocellulosic material is contacted with the aqueous acid solution in step (a) from about 12 hours to about 24 hours.

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5. A method according to claim 1 wherein the lignocellulosic material is contacted with the aqueous acid solution in step (a) under heat.
6. A method according to claim 2 wherein the lignocellulosic material is
5 contacted with the aqueous nitric acid solution in step (a) at a temperature from about 50 °C to below the boiling point of the nitric acid.
7. A method according to claim 2 wherein the aqueous nitric acid solution in
10 step (a) comprises from about 10% to about 30% by weight of the nitric acid.
8. A method according to claim 7 wherein the aqueous nitric acid solution comprises from about 15% to about 30% by weight of the nitric acid.
- 15 9. A method according to 8 wherein the aqueous nitric acid solution comprises from about 20% to about 25% by weight of the nitric acid.
10. A method according to claim 2 wherein the aqueous nitric acid solution comprises from about 10% to about 15% by weight of the nitric acid.
- 20 11. A method according to claim 2 wherein the temperature during the second heating stage of step (b) is from about 73 °C to below 100 °C.
12. A method according to claim 11 wherein the temperature during the second
25 heating stage of step (b) is from about 90 °C to about 95 °C.
13. A method according to claim 2 wherein the temperature during the first heating stage of step (b) is up to about 75 °C.
- 30 14. A method according to claim 13 wherein the temperature during the first heating stage of step (b) is from about 50 °C to about 75 °C.
15. A method according to claim 14 wherein the temperature during the first heating stage of step (b) is from about 60 °C to about 70 °C.
- 35 16. A method according to claim 14 wherein the temperature during the first heating stage of step (b) is from about 50 °C to about 60 °C.

17. A method according to claim 14 wherein the temperature during the first heating stage of step (b) is from about 70 °C to about 75 °C.
18. A method according to claim 2 wherein the first heating stage in step (b) is carried out for at least 15 minutes.
19. A method according to claim 2 wherein the aqueous alkaline solution comprises sodium hydroxide or potassium hydroxide or a combination of sodium hydroxide and potassium hydroxide.
20. A method according to claim 2 wherein the aqueous alkaline solution comprises an amount of alkali solute which is at least the normal equivalent of the nitric acid in the aqueous acid solution in step (a).
21. A method according to claim 2 wherein the aqueous alkaline solution comprises an amount of alkali solute which is at least the molar equivalent of the nitric acid in the aqueous acid solution in step (a).
22. A method according to claim 2 wherein the acid added in step (e) is sulfuric acid.
23. A method according to claim 2 wherein the amount of acid added in step (e) is at least the molar amount of the alkali in the aqueous alkaline solution in step (c).
24. A method according to claim 1 wherein the black liquor is cooled from the temperature in step (c) before the acid is added in step (e).
25. A method according to claim 2 wherein the temperature of the black liquor when the acid is added in step (e) is up to about 75 °C.
26. A method according to claim 25 wherein the temperature of the black liquor when the acid is added in step (e) is from about 5 °C to about 75 °C.
27. A method according to claim 26 wherein the temperature of the black liquor when the acid is added in step (e) is from about 5 °C to about 50 °C.

28. A method according to claim 27 wherein the temperature of the black liquor when the acid is added in step (e) is from about 25 °C to about 50 °C.
- 5 29. A method according to claim 25 wherein the temperature of the black liquor when the acid is added in step (e) is from about 50 °C to about 75 °C.
- 10 30. A method according to claim 1 wherein any aqueous acid solution not absorbed by the lignocellulosic material in step (a) is removed prior to heating the lignocellulosic material in step (b).
- 15 31. A method according to claim 30 comprising collecting any aqueous acid solution which is removed prior to step (b) and recycling the collected aqueous acid solution for use in step (a), and comprising collecting any acid which is distilled off in step (b) and recycling the collected acid for use in step (a).
- 20 32. A method according to claim 1 comprising contacting the lignocellulosic material with water before step (a) to increase the moisture content in the lignocellulosic material.
- 25 33. A method according to claim 1 wherein the starting moisture content of the lignocellulosic material is from about 30% to about 55% by weight of water.
- 30 34. A method according to claim 1 wherein the starting lignocellulosic material comprises undigested lignocellulosic material previously subjected to the method.
35. A method according to claim 1 wherein the starting lignocellulosic material comprises wood chips, wood shavings, sawdust or a combination of two or more wood chips, wood shavings and sawdust.
36. A method according to claim 1 wherein the starting lignocellulosic material comprises pieces of rye, wheat, hemp or a combination of two or more of rye, wheat and hemp.

37. A method according to claim 1 wherein an amber liquor is left following the removal of the lignin, and comprising processing the amber liquor after the lignin has been removed.
- 5 38. A method according to claim 37 wherein the amber liquor is processed to produce unicellular proteins or alcohols or both.
- 10 39. A method according to claim 1 wherein any water which is produced as a reaction byproduct in one or more of the steps is collected and recycled for use in the method.
- 15 40. A method according to claim 2 wherein steps (a) and (c) and the first heating stage in step (b) are each carried out at a temperature from about 50 °C to about 75 °C.
- 20 41. A method according to claim 1 wherein at least one of the heating of the impregnated lignocellulosic material in step (b), the contacting the lignocellulosic material with the aqueous alkaline solution in step (c) and the adding the acid in step (e) is carried out with agitation.
- 25 42. A method according to claim 1 wherein the heating of the impregnated lignocellulosic material in step (b), the contacting the lignocellulosic material with the aqueous alkaline solution in step (c) and the addition of the acid in step (e) are each carried out with agitation.
- 30 43. A method according to claim 1 wherein the method is carried out at atmospheric pressure.
44. A method according to claim 1 comprising washing, pressing, bleaching and drying the pulp removed in step (d).
- 35 45. A method according to claim 1 comprising drying the lignin removed in step (f).
46. A method according to claim 2 wherein step (c) is carried out at a temperature up to about 75 °C.

47. A method according to claim 46 wherein step (c) is carried out at a temperature from about 5 °C to about 75 °C.
48. A method according to claim 47 wherein step (c) is carried out at a temperature from about 50 °C to about 75 °C.
49. A method according to claim 46 wherein step (c) is carried out at a temperature from about 20 °C to about 50 °C.
- 10 50. A method according to claim 47 wherein step (c) is carried out at a temperature from about 30 °C to about 40 °C.
51. A method according to claim 47 wherein step (c) is carried out at a temperature from about 40 °C to about 50 °C.
- 15 52. A method according to claim 1 wherein the lignocellulosic material is immersed in the aqueous acid solution in step (a).
53. A method according to claim 1 wherein the lignocellulosic material is sprayed with the aqueous acid solution in step (a).
- 20 54. A method for producing pulp and lignin from lignocellulosic material, the pulp comprising cellulose, the method comprising:
 - 25 (a) contacting the lignocellulosic material with an aqueous nitric acid solution to impregnate the lignocellulosic material, the aqueous nitric acid solution comprising from about 10% to about 40% by weight of the nitric acid,
 - 30 (b) heating the impregnated lignocellulosic material in two stages, the first heating stage being carried out at a temperature from about 50 °C to about 75 °C for a period of time which is sufficient to depolymerize lignin within the lignocellulosic material without substantially degrading the cellulose or lignin in the lignocellulosic material, the second heating stage being carried out at or above the boiling point of the nitric acid to distill off the nitric acid;

- 5 (c) contacting the lignocellulosic material with an aqueous alkaline solution at a temperature from about 50 °C to about 75 °C to solubilize lignin in the alkaline solution, leaving a black liquor, the aqueous alkaline solution comprising an amount of alkali which is at least the normal amount of the nitric acid in the aqueous acid solution in step (a);

10 (d) removing the pulp from the black liquor;

(e) cooling the black liquor and then adding an acid to the black liquor to acidify the solution to precipitate the lignin;

(f) removing the lignin, leaving an amber liquor; and

15 (g) processing the amber liquor to produce unicellular proteins or alcohols or both,

20 wherein any aqueous nitric acid not absorbed by the lignocellulosic material in step (a) is removed and collected following step (a) prior to heating the lignocellulosic material in step (b) and then recycled for use in step (a), and wherein any nitric acid which is distilled off is collected prior to contacting the lignocellulosic material with the alkaline solution in step (c) and then recycled for use in step (a), and wherein the heating of the lignocellulosic material in step (b), the contacting the lignocellulosic material with the aqueous alkaline solution in step (c) and the addition of the acid in step (e) are each carried out with agitation.

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55. A method for producing pulp and lignin comprising the steps of contacting lignocellulosic material with an acid and, after removing any acid not absorbed by the lignocellulosic material, heating the lignocellulosic material at a temperature up to about 75 °C to effect the acid-catalyzed hydrolytic depolymerization of the lignin in the lignocellulosic material without substantially degrading the cellulose or lignin in the lignocellulosic material, the acid-contacting and heating steps being carried out before the lignocellulosic material is digested in an alkaline liquor, the pulp being removed following the digestion of the lignocellulosic material in the alkaline liquor, the lignin being removed after being precipitated out with

the addition of an acid to the black liquor produced following the digestion of the lignocellulosic material in the alkaline liquor, wherein the pulp comprises cellulose.

- 5 56. A method according to claim 55 wherein the acid-contacting step comprises immersing the lignocellulosic material in an aqueous solution of the acid, and wherein the acid is nitric acid and the aqueous solution comprises from about 10 % to about 40 % by weight of nitric acid.
- 10 57. A method according to claim 56 comprising heating the lignocellulosic material after depolymerizing the lignin but before digesting the lignocellulosic material, at a temperature above the boiling point of the acid in order to distill off the acid.
- 15 58. A method according to claim 44 wherein any liquor that is removed from the pulp by pressing is collected and added to the black liquor prior to adding the acid in step (e).
- 20 59. A method according to claim 45 wherein any liquor which is removed from the lignin during drying is collected and added to the liquor after step (f), and wherein the liquor is processed after the lignin has been precipitated and removed.
- 25 60. The method of claim 1 wherein enough acid is added to the solution in step (e) to lower the pH of the solution to an acidic pH.
- 30 61. A method for treating lignocellulosic material comprising:
- 35 (a) contacting the lignocellulosic material with an aqueous acid solution to impregnate the lignocellulosic material, the aqueous acid solution comprising from about 10% to about 40% by weight of the acid;
- (b) heating the lignocellulosic material in two stages, the first heating stage being carried out for a period of time which is sufficient to depolymerize lignin within the lignocellulosic material without substantially degrading the cellulose or lignin in the lignocellulosic material, the second heating stage being carried out at or above the

boiling point of the acid to distill off the acid, wherein any aqueous acid solution not absorbed by the lignocellulosic material in step (a) is removed prior to heating the lignocellulosic material in step (b);

- 5 (c) contacting the lignocellulosic material with an aqueous alkaline solution under heat to solubilize lignin in the alkaline solution, leaving a black liquor; and

10 (d) removing the pulp from the black liquor, the pulp comprising cellulose.

15 62. A method according to claim 2 wherein the amount of acid added in step (e) is at least the normal amount of the alkali in the aqueous alkaline solution in step (c).

20 63. A method for producing pulp and lignin from lignocellulosic material, the pulp comprising cellulose, the method comprising:

25 (a) contacting the lignocellulosic material with an aqueous nitric acid solution to impregnate the lignocellulosic material, the aqueous acid solution comprising from about 10% to about 40% by weight of the nitric acid;

30 (b) heating the lignocellulosic material in two stages, the first heating stage being carried out for a period of time which is sufficient to depolymerize lignin within the lignocellulosic material without substantially degrading the cellulose or lignin in the lignocellulosic material, the second heating stage being carried out at or above the boiling point of the acid to distill off the acid, wherein any aqueous acid solution not absorbed by the lignocellulosic material in step (a) is removed prior to heating the lignocellulosic material in step (b);

35 (c) contacting the lignocellulosic material with an aqueous alkaline solution under heat to solubilize lignin in the alkaline solution, leaving a black liquor;

(d) removing the pulp from the black liquor;

- (e) adding sufficient acid to the black liquor to precipitate the lignin; and
- (f) removing the lignin from the liquor.

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